



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US99/13033 <b>(22) International Filing Date:</b> 11 June 1999 (11.06.99)  <b>(30) Priority Data:</b> 09/103,923                      24 June 1998 (24.06.98)                      US  <b>(71) Applicant:</b> E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US).  <b>(72) Inventor:</b> KANE, John, Patrick; 113 Gardengate Road, Camden, DE 19934 (US).  <b>(74) Agent:</b> MEDWICK, George, M.; E.I. du Pont de Nemours and Company, Legal Patent Records Center, 1007 Market Street, Wilmington, DE 19898 (US).		<b>(81) Designated States:</b> JP, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> ZINC SULFIDE DELUSTERED FLOCK FIBER  <b>(57) Abstract</b>  A tow used for flock, the tow containing polymer fiber having a denier per filament (dpf) of twenty (20) dpf to less than one (1) dpf, and more preferably, a denier per filament (dpf) of three (3) dpf to less than one (1) dpf. The tow is delustered with from about 0.02 % to about five (5 %) percent, and more preferably from about 0.5 % to about two (2 %) percent, zinc sulfide (ZnS) delusterant by weight. The tow is able to be blade cut to form flock with reduced wear on the cutting blade.		

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ZINC SULFIDE DELUSTERED FLOCK FIBERBACKGROUND OF THE INVENTION

5        Field of the Invention    This invention relates to  
a thermoplastic polymer tow having zinc sulfide (ZnS)  
delusterant therein that is relatively easy to cut to  
form flock fiber, and to the flock fiber formed  
therefrom.

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Description of the Prior Art    Flock fibers are  
very short length fibers that are used to make flocked  
fabric and web products. These products are made by  
adhering flock fibers "on end" to a thin adhesive layer  
15 coated onto a substrate. Usually, during flocking, the  
flock fibers are electrostatically propelled toward the  
suitably prepared substrate. The flock fibers have a  
conductive finish that allows them to align with the  
electrostatic field and to stick substantially  
20 perpendicularly on impact with the adhesive surface.  
Flock fabric has a typical flock fiber density on the  
order of two hundred fifty million (250 million) to  
five hundred million (500 million) fibers per square  
meter.

25        Flock fibers are prepared by a process known in  
the art as "tow conversion". Continuous fibers are  
melt spun, converged and drawn to form a bundle of  
continuous length fibers known as a "tow". Typically  
from about ten to about sixty individual tows, each  
30 having several hundred thousand to over one million  
individual continuous fibers, are assembled into a  
single "massive bundle". This massive bundle is fed  
into a guillotine or rotary cutter. The massive bundle  
is transversely cut by the blades of the cutter into  
35 flock fibers at a rate of several hundred cuts per  
minute. About two billion individual fiber cuts are  
required to form one pound of flock.

The length of the flock fiber is determined in accordance with the diameter of the continuous fiber being cut. As fiber diameter decreases the cut length decreases proportionally. For example, flock cut from  
5 three (3) denier continuous fiber has a length of about 0.063 inch (1.60 mm). Flock cut from 1.5 denier nylon 6,6 fiber [with a diameter of 0.0005 inch (0.0166 mm)] has a length of about 0.050 inch (1.27 mm). Thus, the number of cuts per pound of flock increases as the  
10 length of the fiber decreases.

Sharp cutting blades are critical to the production of quality flock. Very sharp blades produce flock with clean cut ends. Cleanly cut ends allows the individual flock fibers to separate readily. As a  
15 cutter blade wears it loses its sharp edge and it is no longer able to make clean transverse end cuts. Instead, the ends of the flock are smeared or fused together. A person skilled in the art of commercial tow conversion is able to judge when an objectionable  
20 level of fused fiber in the flock product exceeds flock mill standards and requires a process interruption. Excessively fused fibers result in poor quality flock, poor flocking performance, waste, and off-quality flocked fabrics.

25 Blade edge retention is especially critical in forming flock from relatively small diameter, finer denier fibers. The fine denier fibers fuse more easily and are less tolerant to worn blade edges than fibers of larger diameter (denier).

30 Flock cutter blade honing and replacement are known expedients to reestablish a worn blade edge and to improve flock cutting performance. Blade honing is usually done by hand. A given blade may be re-honed only a limited number of times (typically on the order  
35 of ten to twelve times) before the blade must be replaced. However, either honing or replacement is a time consuming interruption to the tow conversion process. The time interval between blade honing or

replacement is critical to the efficiency of the tow conversion operation.

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5       A delusterant is a additive that opacifies polymer to produce a fiber having a more matte appearance with suppressed glitter. For primarily economic reasons the most common pigment used as a delustrant is anatase titanium dioxide ( $\text{TiO}_2$ ). The anatase titanium dioxide  
10 is usually added in amounts from about 0.02% to about five (5%) percent by weight of the fiber. Anatase titanium dioxide has a relatively high light scattering power, with a refractive index of 2.55. However, anatase titanium dioxide is a relatively hard material,  
15 having a hardness measured on the mohs scale of 5.5 to 6.0.

Other delusterants, such as zinc sulfide, are also known in the art. Compared to anatase titanium dioxide zinc sulfide is a more lubricious and a softer  
20 material, having a hardness on the mohs scale of about 3.0. However, since zinc sulfide has a larger optimal particle size and a lower index of refraction than anatase titanium dioxide, its light scattering ability is less. Quantitatively, the light scattering ability  
25 of zinc sulfide is on the order of about seventy (70%) percent to seventy-five (75%) percent that of anatase titanium dioxide. It would appear to follow that for a given denier per filament, comparable luster may be achieved only if the amount of zinc sulfide in the  
30 fiber is significantly greater than the corresponding amount of anatase titanium dioxide.

It has been recognized in the art that when fiber delustered with anatase titanium dioxide is used to make flock, wear on the cutting blade increases and the  
35 time interval between blade honing or replacement is concomitantly significantly reduced. The observed increase in blade wear is believed due to abrasion by delusterant grains within the volume of the filament on

the blade edge. As earlier noted this excessive blade wear is detrimental to the efficient production of flock from fine denier delustered fibers.

5 In other areas of the fiber production art, such as the production of staple-length fibers used for carpet yarn, the problem of guide wear due to abrasion with delustered fiber is addressed and reduced through the application of a topical yarn finish. However, the expedient of a topical finish may not be effective for  
10 fibers intended for tow conversion. In some instances the tow converter scours the tow prior to cutting to remove any finish. However, even if the tow is not scoured, the surface finish does little to protect the blade from abrasion by particles located in the  
15 interior of the fiber.

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In view of the foregoing it is believed advantageous to provide a fibrous tow which is able to  
20 be cut easily and with less wear effects on cutter blade edges, and which produces flock exhibiting luster properties similar to a tow having anatase titanium dioxide therein.

#### 25 SUMMARY OF THE INVENTION

The invention is directed to a delustered thermoplastic polymer tow formed from a plurality of fibers, wherein each fiber has from about 0.02% to about five (5%) percent, and more preferably from about  
30 0.5% to about two (2%) percent, zinc sulfide delusterant by weight.

Each fiber has a denier per filament (dpf) in the range from about twenty (20) dpf to less than one (1) dpf. The invention may be most preferably used with  
35 fibers having a denier per filament (dpf) in the range from about three (3) dpf to less than one (1) dpf. The present invention may also be used for fibers having a

denier per filament (dpf) in the range of about fifteen (15) dpf to about eighteen (18) dpf.

In another aspect the invention is directed to flock produced by the conversion of such tow.

5 It has been found that tow in accordance with the present invention is susceptible to blade cutting to form flock with reduced wear on the cutting blade. The time interval between honing or replacement of cutter blades used to convert fibrous tows delustered with  
10 zinc sulfide is dramatically increased in comparison to fibrous tows delustered with anatase titanium dioxide.

#### BRIEF DESCRIPTION OF THE DRAWINGS

The invention will be more fully understood from  
15 the following detailed description thereof, taken in connection with the accompanying drawings, which form a part of this application and in which:

Figures 1A and 1B form a stylized schematic representation of a split spinning, drawing and packing  
20 arrangement for producing a thermoplastic polymer tow having fibers with zinc sulfide delusterant therein in accordance with the present invention; and

Figure 2 is a stylized schematic representation of an arrangement for converting thermoplastic polymer tow  
25 in accordance with the present invention into flock.

#### DETAILED DESCRIPTION OF THE INVENTION

Throughout the following detailed description similar reference numerals refer to similar elements in  
30 all Figures of the drawings.

With reference to Figure 1A shown is a schematic representation of an arrangement for spinning a thermoplastic polymer tow having zinc sulfide delusterant therein in accordance with the present  
35 invention. The input of an extruder 10 is connected by a line 12 to a source (not shown) of a thermoplastic base polymer. The thermoplastic base polymer may be introduced into the extruder 10 in flake form, in which

event the polymer source connected to the input line 12 is a gravimetric feeder of polymer pellets.

Alternatively, the line 12 may take the form of a transfer pipe which carries molten polymer from a continuous polymerizer or from another extruder.

Any thermoplastic polymer material, such as polyamide, polyester and polypropylene, may be used. However, the invention is most preferably applicable to polyamides (nylon 6,6, nylon 6, nylon 4,6, nylon 6,10, nylon 6,12, nylon 7), copolyamides, polyesters (polyethylene terephthalate, polytrimethylene terephthalate, polybutylene terephthalate, polyethylene naphthalate, polytrimethylene naphthalate, polybutylene naphthalate) and copolyesters.

It has been found impractical to add zinc sulfide directly to the polymer salt while in the autoclave or in the continuous polymerizer. Therefore, in order to avoid objectionable decomposition the zinc sulfide additive is introduced into the extruder via a masterbatch of pigment in a carrier resin. The carrier resin is compatible with the base thermoplastic polymer being produced. The masterbatch zinc sulfide particles from a hopper 14 are conveyed through a line 16 and fed into the extruder 10. The masterbatch contains from about ten (10%) to about fifty (50%) percent by weight zinc sulfide. Suitable zinc sulfide particles are sold by Sachtleben Chemie, Duisberg, Germany, under the trademark Sachtolith® HDS.

The masterbatch is fed into the base polymer, mixed and dispersed in the extruder 10. The polymer mixture from the extruder 10 is pumped by a pump 20 through transfer lines 22 to an array of fiber spinning positions 24. Although four are illustrated it should be understood that any convenient number of such positions 24 may be used to constitute the array. The continuous fibers 26 emanating from each position 24 are converged into a threadline 28. The threadlines 28



from each spinning position 24 are gathered into a tow 30. The tow 30 is dispensed into a can 32.

The can 32 from the array of fiber spinning positions 24 is next moved to a drawing and tow packing apparatus schematically illustrated in Figure 1B. The tow 30 is fed by a feed section 36 to a draw section 38. In the draw section 38 the fibers in the tow 30 are drawn to desired denier per filament. One or more tow(s) 30 from additional can(s) 32 may be ganged into the drawing and tow packing apparatus, as is indicated at 40. The tow 30 is packaged in a carton 42 for shipment to a tow converter for conversion into flock.

Although Figures 1A and 1B illustrate a split spin-draw arrangement, a typical textile continuous spin/draw or a spun orientation process that winds fiber ends onto a bobbin may be used. Up to several thousand bobbins may be creeled together to form a tow in an operation known in the art as "beaming" or "ball warping".

However formed, the tow 30 and each continuous fiber 26 therein contains zinc sulfide delusterant in the range from about 0.02% to about five (5%) percent by weight of the spun fiber. More preferably, zinc sulfide delusterant is in the range from about 0.5% to about two (2%) percent by weight of the fiber.

The fibers 26 forming the tow 30 may have a denier per filament (dpf) in the range from about twenty (20) dpf to less than one (1) dpf (the so-called "micro-fiber" or "sub-denier" range. The invention is most preferably used with finer denier fibers, that is, fibers having a denier per filament (dpf) in the range from about three (3) dpf (so-called "fine" denier fiber) to fibers having a denier per filament of less than one (1) dpf (so-called "micro-fiber" or "sub-denier" fiber). Typically, sub-denier fiber can have a dpf in the range from from 0.9 to 0.4 or less. The invention may be also used for heavier or "bulk" fibers

having a denier per filament (dpf) in the range of about fifteen (15) dpf to about eighteen (18) dpf.

Figure 2 is a schematic illustration of the operative elements of a tow conversion apparatus for cutting the tow 30 produced by the arrangement of Figures 1A and 1B.

As suggested in Figure 2 the tows 30 from about ten (10) to about sixty (60) cartons 40 (Figure 1B) are assembled into a massive bundle diagrammatically indicated by the reference character 44. The massive bundle 44 is presented to a flock cutter apparatus generally indicated by the reference character 48. The cutter 48 may be implemented in the form of a guillotine cutter (as illustrated) or a rotary cutter, as desired. The massive bundle 44 is introduced into the nip 48N defined between a flying knife 48F and the spaced bed knife 48B.

As the flying knife 48F moves toward the bed knife 48B in the direction of the reciprocating arrow 48A the fibers 26 forming the bundle 44 are cut into flock filaments 50. As seen in the enlargement, each flock filament 50 has a predetermined length dimension 50L and a diameter dimension 50D dependent upon the denier of the fibers 26 forming the bundle 44.

As set forth earlier, it has been observed that the use of fibers delustered with anatase titanium dioxide to make flock result in increased wear on the blades of the cutter 44 and a concomitantly significant reduction in the time interval between blade honing or replacement. The problem of excessive blade wear is especially detrimental to the production of flock from fine denier, delustered fibers.

It has been found that fiber and tow delustered by zinc sulfide in accordance with the present invention is able to be cut into flock with reduced wear on the cutting blade. As a result cutting blades perform at acceptable levels for longer periods of time as compared to fiber delustered with anatase titanium

dioxide. The following Examples show the time interval between blade honing or replacement is approximately doubled.

5

### Examples

The examples illustrate the improvement in cutting performance achieved with nylon 6,6 tow delustered with zinc sulfide (ZnS) in accordance with the present invention as compared to a nylon 6,6 tow delustered with anatase titanium dioxide (TiO<sub>2</sub>).

#### Example 1. Comparative

A nylon 6,6 tow of 1.5 dpf fibers was produced with 1.5 weight percent anatase titanium dioxide delusterant. A massive bundle having approximately 7.3 million individual 1.5 dpf fibers was assembled and fed to a cutter for conversion to flock. Flock of 0.050 inch length (1.27 mm) was produced at a predetermined cut speed. The flock had a fully delustered appearance, as established by direct visual observation by a person skilled in the art. A person skilled in the art established the need for blade honing due to wear by direct observation of the number of fused fibers in the flock. Flock production was interrupted while the blade was honed to original sharpness. The time interval between such production interruptions was recorded for a period of several days. The average time interval between such production interruptions was normalized to 1.00 time-units.

#### Example 2. The Present Invention

A nylon 6,6 tow identical to that of Example 1, except for 1.5 weight percent zinc sulfide delusterant in the polymer, was spun, drawn and assembled. The zinc sulfide was added to the base polymer by a masterbatch containing about forty (40%) percent by weight Sachtolith® HDS zinc sulfide particles, with

the remainder being nylon 6 carrier resin. The zinc sulfide particles, compounded with the carrier resin by the manufacturer, were obtained from Sachtleben Chemie, Duisberg, Germany. Tow conversion to flock was carried out in a fashion identical to Example 1. The resulting 1.5 dpf flock was produced with a 0.050 inch (1.27 mm) cut length. The number of filaments cut, cut speed, and cut length, and blade life evaluation method were identical to Example 1. The flock exhibited the same fully delustered appearance as the flock of Example 1, as determined by visual inspection by the same skilled artisan. The time interval between production interruptions was again recorded for the same period of days. The average time interval between such process interruptions with the zinc sulfide delustered fiber of the present invention increased to 1.96 time-units (normalized).

**Conclusions:** The foregoing comparative examples clearly show that a fiber delustered using zinc sulfide (ZnS) in accordance with the present invention is susceptible to blade cutting to form flock with reduced wear on the cutting blade. The fiber of the present invention offers superior cutting performance over fibers delustered with anatase titanium dioxide ( $\text{TiO}_2$ ). Blade edge wear is significantly reduced with the softer, less abrasive, zinc sulfide pigment as the delusterant, as evidenced by the beneficial increase in the time interval between blade honings (1.96 time-units to 1.00 time units). Higher tow conversion mill productivity yields and flock product superior in cut quality are thus obtained.

It is also noted from the examples that the amount of zinc sulfide was the same (1.5%) as the amount of titanium dioxide. However, given the diminished index of refraction of zinc sulfide, for a given denier per filament and a given luster level, it would be predicted that the amount of zinc sulfide required to

deluster a fiber would be significantly greater than the amount of titanium dioxide required to deluster a comparable fiber. Quantitatively, if 1.5% by weight titanium dioxide imparts a predetermined luster level to a fiber of a predetermined dpf, it would have been expected that a level of about 2.2% to about 2.5% by weight zinc sulfide would be needed to impart the same luster level for a fiber of the same dpf.

Surprisingly, however, the amount of zinc sulfide was significantly below expectation.

What is claimed is:

1. A delustered thermoplastic polymer tow  
5 formed from a plurality of fibers wherein each fiber  
has from 0.02% to five (5%) percent zinc sulfide  
delusterant by weight and wherein each fiber has a  
denier per filament (dpf) in the range from twenty (20)  
dpf to less than one (1) dpf,  
10 whereby the tow is susceptible to blade cutting to  
form flock with reduced wear on the cutting blade.
2. The delustered thermoplastic polymer tow of  
claim 1 wherein each fiber has from 0.5% to two (2%)  
15 percent zinc sulfide delusterant by weight.
3. The delustered thermoplastic polymer tow of  
claim 1 wherein each fiber has a denier per filament  
(dpf) in the range from three (3) dpf to less than one  
20 (1) dpf.
4. The delustered thermoplastic polymer tow of  
claim 1 wherein each fiber has a denier per filament  
(dpf) in the range from eighteen (18) dpf to fifteen  
25 (15) dpf.
5. The delustered thermoplastic polymer tow of  
claim 1 wherein the thermoplastic polymer is a  
polyamide.  
30
6. The delustered thermoplastic polymer tow of  
claim 1 wherein the thermoplastic polymer is a  
polyester.
- 35 7. Flock formed from a thermoplastic polymer  
fiber, wherein the fiber has from 0.02% to five (5%)  
percent zinc sulfide delusterant by weight and wherein

the fiber has a denier per filament (dpf) in the range from twenty (20) dpf to less than one (1) dpf,

whereby the fiber is susceptible to blade cutting to form flock with reduced wear on the cutting blade.

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8. The flock of claim 7 wherein the fiber has from 0.5% to two (2%) percent zinc sulfide delusterant by weight.

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9. The flock of claim 7 wherein the fiber has a denier per filament has a denier per filament (dpf) in the range from three (3) dpf to less than one (1) dpf.

10. The flock of claim 7 wherein the fiber has a  
15 denier per filament (dpf) in the range from eighteen (18) dpf to fifteen (15) dpf.

11. The flock of claim 7 wherein the thermoplastic polymer is a polyamide.

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12. The flock of claim 7 wherein the thermoplastic polymer is a polyester.

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 99/13033

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 D01F1/04 D01F6/60 D01F6/62

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 D01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 356 521 A (BOLTNIEW A) 5 December 1967 (1967-12-05) the whole document ---	1-12
A	CH 661 741 A (INVENTA AG) 14 August 1987 (1987-08-14) column 2, line 50 - line 53; claims ---	1-12
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☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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Patent document cited in search report		Publication date	Patent family member(s)	Publication date
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